Validated Raman Spectral Libraries as a Resource for Chemical Threat Identification

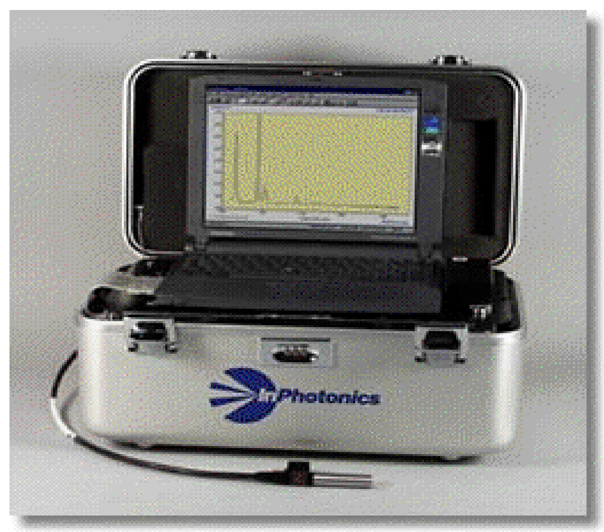
NIST scientists are developing reference libraries of chemical spectral signatures to enable the use of Raman spectroscopy in homeland security applications. Raman spectroscopy is a measurement technology capable of identifying chemicals through the walls of their containers.

S. J. Choquette (Div. 831)
B. A. Benner (Div. 839)

NIST is developing validated Raman spectral libraries for homeland defense applications. Raman spectroscopy is an attractive analytical technique for the identification of unknown chemical compounds because it requires no sample preparation, can be used through common transparent sample containers, and with current technological innovations, is very portable. However because Raman is an emission process, each instrument has a unique response function. As a result, Raman libraries of chemical compounds are currently vendor-specific and not transportable between systems. Since FY 2000 ACD has been involved with the development of instrument performance standards (SRM's) to correct for instrument response, and recently has been funded by DHS to develop an instrument corrected Raman spectral database. Because the Raman spectrum of a neat compound is an intrinsic property of the material, this primary data activity should lead to a standardized, universally useable, Raman database. This years' activities have involved the initial development of a validated spectral library of Toxic Industrial Compounds (TICs) and the writing of an ASTM practice guide for determining the resolution of a Raman spectrometer.

The NIST Raman Spectral Library of toxic industrial chemical spectral signatures is being developed as a resource to improve chemical detection in the homeland security and environmental remediation sectors.

Nineteen compounds were chosen from a list of over 350 complied by the US EPA. TICs are industrially important compounds that are routinely manufactured and transported in every major industrial country. Typically they are less toxic than military chemical warfare agents, but are highly reactive and much easier to obtain. The initial list comprised families of isocyanates, formates, and compounds with unsaturated double bonds. Each was assessed for purity using GC-FID and the identity confirmed with GC-MS. Several compounds were too thermally unstable for GC analysis and an NMR technique is under development to assess the purity of these materials. In addition, several reaction products, in excess of 5% were discovered during the course of the purity assessment, either as impurities or reaction products with air or the acidic protons of the solvents used in the GC analysis. These reaction products will also be included in the library as they would be likely present when the material is used in an industrial setting. Instrument corrected Raman spectra of each of the compounds were acquired with 785 nm and 1064 nm laser excitation for the compounds in their original containers (amber bottles) and also in quartz sample cells. The amber bottle typically used for reagent grade materials effects the shape of the spectra and should be included in the library.



Development of lasers, filters, and detectors by the telecommunications industry enables use of single stage spectrographs for Raman Spectroscopy. Portable Raman is commercially available.

To support our FY 2005 work on algorithms, a practice guide for determining the resolution of a Raman spectrometer was written and presented for ballot to the ASTM E 13.08 subcommittee for Raman spectroscopy. The practice guide will enable the end user of a field portable system to assess the resolution of their instrument using either pen lamps or the calcite spectrum. This information will be used in conjunction with NIST standards (SRM 2241) to calculate instrument corrections for spectra acquired using these field portable systems. This practice will facilitate searching end user generated spectra on a NIST database.

Raman Spectroscopy provides sample identification through common containers, in real-time and without sample preparation.

Future plans: We plan to develop a web-based version of the library (NIST CHEMWEB book) that will enable users to compare their spectra with NIST's. In addition FY 2006 we will work on writing a standard practice guide for the use of our intensity standards for the acquisition of instrument corrected Raman spectra.

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